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Physical characterization of a helium-oxygen-acetylene flame supported on a water-cooled burner has shown the flame to possess a high degree of laminarity and a maximum safe operating temperature of 2760 K (with 60% helium diluent). Safe operating conditions for the flame have been established to enable its safe and flashback-free operation. In addition, the spectral background of the flame has been examined and found to be relatively low compared to other high temperature flames, suggesting its use as a promising

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alternative atom cell for atomic emission, absorption, or fluorescence
spectrometry.

Physical Characterization of a
New Atom Cell for Atomic Spectrometry:
The Helium-Oxygen-Acetylene Flame

by

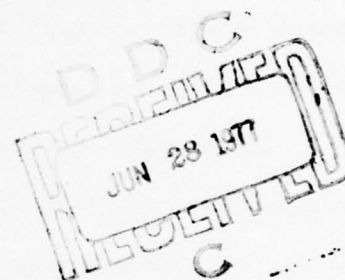K.A. Saturday and G.M. Hieftje

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ANALYTICAL CHEMISTRY

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Functioning as a combined atom source, sample cell, and excitation medium for atomic spectrometry, the typical analytical flame forms an environment which has a complex and dynamic yet easily utilizable character unparalleled by any other analytical source. In this application, the flame serves a number of functions essential to flame spectrometric analysis, including nebulization of the sample solution, desolvation of the resulting aerosol, vaporization of solute particles and atomization of the vaporization products. Unfortunately, flames are notoriously inefficient and poorly understood in how they accomplish these results and only through a basic understanding of how each step is accomplished will the flame's utility be improved markedly over that now attainable.

In the past, improvements in flames were derived largely through empirical means. Variations in flame-gas constituents and their proportions were found to be most effective in improving the flame, although burner design, the use of sheathing gases, and the development of different flame shapes were also found to be important. However, because of the complex environment within each flame and the interrelationships of variables affecting that environment, it was difficult or impossible to single out individual flame properties for optimization. Clearly, what would be desirable would be to separate all variables, characterize them, and employ the results of such characterization to generate improvements. In our laboratory, just such investigations have been underway for some time, and the present report indicates the utility of those findings in developing new and improved flames for use in spectrometric analysis.

In recent studies (1-3), we investigated the processes of droplet desolvation and solute particle vaporization in an analytical flame. These studies have yielded quantitative relationships describing those processes and have increased our knowledge of atom formation to the point where some meaningful changes could be made to increase the flame's performance. In a study on the mechanism

of droplet desolvation in a flame (1,3), it was found that flame-gas thermal
conductivity played an important role in the desolvation process. A subsequent
study demonstrated that replacement of the air component in an air-acetylene
flame with a high thermal-conductivity oxidizer gas mixture (helium + oxygen)
increased the aerosol desolvation and sample utilization efficiencies (3). Un-
fortunately, with these advantages came an increased occurrence of flashback,
thermal deformation of the burner components, and the high cost of the helium
diluent gas (3,4).

Besides its increased sample utilization and atom formation efficiencies,
the helium-oxygen-acetylene flame holds the possibility of increased fluores-
cence yield because of its low concentration of quenching species. Unlike
most analytical flames, the helium-oxygen-acetylene flame has as its primary
constituent a monatomic gas (helium), which has an unusually low collisional
cross section (5). Helium-diluted hydrogen-oxygen flames have already been
shown to exhibit higher quantum efficiencies than similar nitrogen-diluted
flames (6); contrasted to these previously studied flames, the helium-oxygen-
acetylene flame should produce higher atom concentrations, because of the
large number of electrons and carbon-containing radicals it produces (7).
To offset this advantage somewhat, the acetylene-fueled flame would be expected
to exhibit greater quenching than a similar hydrogen-fueled flame.

The present study seeks to increase the utility of the helium-oxygen-
acetylene flame by developing a burner system to produce a stable, reproducible
flame and to establish safe operating conditions for the new system. Physical
characteristics of the resulting flame system were investigated in order to
compare the new flame with others commonly employed. Laminarity of the flame
was investigated using Schlieren photography, and temperature profiles of an
analytically useful flame were obtained. For practical applications, the

background spectrum of the new flame was compared to that of a commonly used
air-acetylene flame.

EXPERIMENTAL

The burner and gas flow system used with the $He-O_2-C_2H_2$ flame are des-
cribed in this section. In addition, special techniques used to characterize
the flame are discussed.

Burner. A modified version of the Meker-type burner described by
Hieftje (8) was used to support the new flame. The burner top, as shown
in Figure 1, contained 169 exit ports of 0.45 mm diameter, which were arranged
in concentric rings. Port diameter was chosen on the basis of a tradeoff
between safety and convenience in operation and fabrication; the 0.45 mm
diameter ports are small enough to prevent flashback but large enough for
easy machinability and for avoidance of clogging by combustion products and
sample residue. Silver-soldered to the burner top was a single turn of
copper tubing (5 mm I.D.) through which tap water was passed at 0.5 ℓ/min.
At this flow, the cooling coil absorbed sufficient heat from the burner top
to eliminate thermal deformation of the burner and to further reduce the
likelihood of flashback.

Flame Gases. As shown in Figure 2, purified helium, oxygen (Matheson
Co., Joliet, Ill.) and acetylene (Linde Div., Union Carbide Corp.; New York,
N.Y.) were supplied through single-stage regulators (Matheson Co.) and measured
with dual-float rotameters (He: Matheson Co.; O_2 and C_2H_2: Dwyer Manufac-
turing Inc., Michigan City, Ind.). The calibration of each flowmeter was
established by a wet-test meter (Precision Scientific Co.; Chicago, Ill.).
Needle valves and toggle shut-off valves (Nupro Co., Cleveland, Ohio) were

used to control the gas flows. After exiting from their flowmeters, the helium and oxygen were mixed at a tubing "Y" and fed through a coiled tube (4) to ensure good mixing before the gases entered the burner chamber. The acetylene was fed directly to the burner from the flow system. When an air-acetylene flame was employed, the air (Matheson Co.) was also fed directly to the burner from the rotameter (Dwyer Manufacturing inc.). All flows reported in this paper have been corrected to STP.

Determination of Safe Operating Conditions. The limits of safe operation of the new burner and flame were ascertained by determining the flame gas flows necessary to prevent flashback. To perform these measurements, a stable flame was established which met the desired requirements of fuel-to-oxidant flow ratio or oxygen flow rate. Small changes in the helium flow were then made until flashback occurred. After each decrement in helium flow, thermal equilibrium was allowed to establish at the burner top before further flow adjustments were made. Thermal equilibrium was judged on the basis of a stable reading from a chromel-alumel thermocouple attached to the burner head. After each flashback was incurred, flow readings were taken. Flashback flow measurements were taken several times for each set of flame conditions to insure reproducibility of the data.

Schlieren Photography. Schlieren photographs (9) were produced using two spherical mirrors designed for that purpose and obtained from Edmund Scientific Co., Barrington, N.J. A pin-hole light source for the photographs was constructed by focussing the tungsten-halogen lamp from a 35 mm slide projector (model AF Carousel, Kodak) onto an iris diaphragm which was placed at the focus of the first spherical Schlieren mirror. The collimated beam produced by the first mirror was passed through the flame to a second spherical mirror which focussed the light at a circular aperture (10). Only

those light rays which remained unrefracted as they passed through the flame were allowed to pass through the circular aperture and to the focal plane of a photographic film holder placed behind the aperture. Further details about the Schlieren technique can be found in the literature (9, 10).

Flame Temperatures. Flame temperatures were measured using the sodium line-reversal technique (11). In these measurements, a tungsten strip lamp served as the primary source and was calibrated with an optical pyrometer (Pyrometer Instrument Co., Northvale, N.J.) whose validity is traceable to the National Bureau of Standards. To further assure accuracy, the pyrometer was periodically checked with an NBS-traceable tungsten-filament lamp (No. 18AT10/2, General Electric Co., Cleveland, Ohio). Detection of the reversed condition (indicating equality of the lamp and flame temperatures), as described by Snelleman (11), was simplified by use of a wavelength-modulated monochromator and synchronous detection system (12).

RESULTS AND DISCUSSION

The Determination of Safe Operating Limits: Flashback Studies

Results of an earlier study (3) have shown the He-O_2-C_2H_2 flame to be flashback-prone when supported on a burner designed for operation with an air-acetylene mixture. This finding is not surprising if one considers the factors affecting the formation of a pre-mixed, laminar flame. The stabilization of such a flame on a burner top is governed by three processes: first, the flame gases are consumed at a rate (characterized by their burning velocity) which is directly related to the composition of the flame gas mixture; second, the walls of the burner ports exert a quenching effect on the combustion of the gas mixture, making the burning velocity zero near the port walls; third, a

short distance from the port walls, the flame gases again attain their characteristic burning velocity (9,13).

For a gas exhibiting laminar flow out of a burner port, a Poiseuille velocity distribution exists across the port diameter. To prevent flashback, the exiting gas velocity at every point in the burner port must be greater than or equal to the backward-propagating burning velocity at that point. The gas flow which produces a velocity profile which is everywhere just equal to or greater than the burning velocity is said to generate the critical boundary velocity gradient. This flow is then the lowest which will prevent flashback.

Flashback occurs when the gases flowing from the burner port do not exceed the critical boundary velocity gradient, causing the flame to propagate into the burner's mixing chamber and detonate the gas mixture there. Obviously, the larger the burner port or the greater the burning velocity of the gas mixture, the greater will be the gas velocity or flow required to prevent flashback. Because the $He-O_2-C_2H_2$ mixture has a far greater burning velocity than air-acetylene, it is not suprising that the former flame could not be safely supported on a burner designed for air-acetylene.

The general approach to producing a safe and flashback-free flame can take two courses: the burner ports can be made small enough to extend the quenching effects of the walls throughout the port, or the minimum gas velocity required to maintain a safe flame can be determined for a particular burner port size. The first of these courses is often not practicable for flames of the kind being studied here. In order to calculate the port diameter which is small enough to maintain the flame under the worst possible conditions, a knowledge of the combustion characteristics for all possible combinations of gas mixture must be available. When a burner port exhibits this behavior, it is said to be at or below the quenching diameter for the gas mixture(s) used. Quenching diameter

values for several flames have been calculated or empirically determined (8, 14,15). Unfortunately, for rapidly burning gas mixtures, the required quenching diameters are often very small. For example, an oxygen-acetylene mixture has a quenching diameter of 0.406 mm (15). Such small ports are generally imprac- tical because of the difficulty of machining a multiport burner head and the likelihood of clogging of the ports with nebulized solution and combustion products. In this situation, the second approach must be taken and safe oper- ating conditions for the flame must be empirically determined. This latter method is the one ordinarily adopted by instrument manufacturers who offer burners for nitrous oxide-acetylene flames.

The critical boundary velocity gradient for differing flame compositions depends not only on the burning velocity of the flame gases but also on their density, thermal conductivity and specific heat (16). For a flame composed of gases whose composition is either pure (i.e., C_2H_2) or constant (i.e., air), the burning velocity has been shown to change relatively little over a wide range of fuel/oxidant ratios, when compared with the large changes in burning velocity which occur when varying amounts of a diluent gas are added to the same gas mixture (9,17). Therefore, varying the percentage of diluent gas in a flame gas mixture is one method of controlling the burning velocity of the flame. For instance, an acetylene flame supported by a 21% O_2 mixture with N_2 has been shown to have a burning velocity one-tenth that of a flame supported by pure O_2 (17). Similar studies on other hydrocarbon flames have shown a four-fold increase in burning velocity when helium was substituted for nitrogen as a diluent in the oxidizer gas (18). For all mixtures it was found that the burning velocity decreased as the fraction of diluent increased.

The amount of helium required in the present study to maintain a stable flame for various acetylene/oxygen flow ratios (i.e., fuel/oxidant ratios)

is shown in Figure 3. To obtain the data in Figure 3, the oxygen flow was maintained at 4.1 ℓ/min; the variation in fuel/oxidant was accomplished by changing the acetylene flow rate. The slight changes in burning velocity of the mixture so produced were countered by altering the amount of helium diluent gas required to prevent flashback. Interestingly, the total gas flow reuqired to prevent flashback was found to remain nearly constant at 11.5 ℓ/min between the fuel/oxidant ratios of 0.45 to 0.87, with slightly lower total flows required at the low fuel/oxidant ratios.

The data displayed in Figure 3 follow a predictable pattern. At the stoichiometric gas-flow ratio (fuel/oxidant = 0.40), the greatest percentage of the oxygen and acetylene components react in the combustion process, to produce a maximal burning velocity. However, air entrainment and in-complete combustion result in a shifting of this maximum burning velocity point to slightly richer measured gas mixtures. At fuel/oxidant ratios away from stoichiometric, the gas burning velocity decreases, as signalled by the smaller percentage of helium required to prevent flashback. This behavior suggests that portions of the acetylene or oxygen flows are not actively involved in the combustion process and act as diluent gases.

Figure 3 reveals that the helium content of the He-O_2-C_2H_2 flame gas mixture must be nearly fifty percent to prevent flashback at all values of fuel/oxidant ratio. In addition, if the oxygen flow is changed, the helium requirement is also altered as shown in Figure 4. To construct Figure 4, a constant fuel/oxidant ratio of 0.49 was chosen to represent the region of greatest helium requirement (cf. Figure 3); also, oxygen and acetylene flows were reduced simultaneously in order to maintain a fixed fuel/oxidant ratio. Thus, the increase in oxygen flow (horizontal axis of Figure 4) also represents

an increase in the total flow of active components in the gas mixture.

From Figure 4, lower oxygen and acetylene flow rates require correspondingly greater helium to prevent flashback. It might then be expected that a constant total flow (including helium) for all oxygen/acetylene combinations would be required. However, Figure 5 shows that the total required flow decreases monotonically with oxygen flow; obviously, the increased dilution of the combustible gases by helium reduces the mixture's burning velocity.

Useful operating limits of the helium-oxygen-acetylene flame are delineated in Figure 6. Because the fuel/oxidant ratio of 0.49 represents the greatest demand for helium (cf. Figure 3), the percent helium required for a specific total flow at this fuel/oxidant ratio will suffice for any other fuel/oxidant ratio. Of course, other fuel/oxidant ratios will permit the use of flame gas mixtures with lower helium content. The lowest helium demand is at the extremes of the useful fuel/oxidant range which extends between the point at which the flame turns luminous (fuel/oxidant = 0.97) and where the acetylene flow becomes too low to be accurately monitored (fuel/oxidant = 0.25). At these fuel/oxidant ratios the helium demand is 28% and 45%, respectively. However, as a general rule, it can be ascertained from Figure 6 that a typical analytical flame (7 ℓ/min total flow or more), supported on a burner head with 0.45 mm diameter ports, will operate safely with a mixture of helium, oxygen and acetylene containing 55% or more helium.

Flame Laminarity: Schlieren Studies

Schlieren methods have long been used to observe spatial refractive index changes within flames and other combustion sources (9,10). Information obtained from Schlieren photographs has been used to correlate turbulence in the flame background radiation to noise seen in an atomic absorption measurement (19). In the present study, the laminarity of the helium-oxygen-acetylene flame was investigated using Schlieren methods.

The Schlieren image produced by the helium-oxygen-acetylene flame (Figure

7A) is identical to that of an air-acetylene flame which was found by others
to exhibit a high degree of laminarity (8,11,20). A slight turbulence at the
edges of the flame and in its upper regions observable in Figure 7A is caused
by the entrainment of air. However, the central portion of the flame remains
fairly laminar throughout the most commonly used region (within 10 cm above
the burner top).

For comparison, the Schlieren image of a Bunsen flame is shown in Figure
7B. The regions of turbulence within the Bunsen flame produced by the mixing
of flame gases and air are easily observed in the high-contrast regions of the
image (9). The wide degree of refractive index change in the mixing gases
produces the sharp contrasts.

Flame Temperatures

The importance of flame temperature to practical flame spectrometry has
long been known. The use of the nitrous oxide-acetylene flame illustrates
the increase in sensitivity and decrease in matrix interferences that higher
flame temperatures can bring, but also betrays the effects of increased ioni-
zation (21). Recent studies have shown the important role flame temperature
plays in the desolvation and vaporization processes in flames (1-3).

The change in $He-O_2-C_2H_2$ flame temperature with fuel/oxidant ratio at
two different heights in the flame is shown in Figure 8. To produce Figure 8,
the total gas and oxygen flow rates were kept constant, but the acetylene and
helium flow rates were varied oppositely to produce the desired fuel/oxidant
ratio and total flow. The results show a rise in temperature with increasingly
richer flames and decreasing helium content.

From Figure 8, the $He-O_2-C_2H_2$ flame exhibits temperatures which are higher
than those of an air-acetylene flame $(2360°K)$ (4) and approach those of a
nitrous oxide-acetylene flame $(2750°K)$ (4). The flame also offers the ability

to vary the temperature over a 200-300°K range by careful choice of fuel/ oxidant ratio and helium content. This ability enables an appropriate temperature and flame composition to be selected independently for the most sensitive determination of any element.

A vertical temperature profile of the helium-oxygen-acetylene flame is shown in Figure 9. The flame composition used to produce Figure 9 was that corresponding to the highest temperature displayed in Figure 8 (i.e., fuel/ oxidant = 0.85). A nearly linear temperature decrease occurs with height in the flame, with a gradient of approximately 34°K per centimeter from the burner top.

Background Spectra

A comparison between the background spectrum of the helium-oxygen-acetylene flame and that of an air-acetylene flame with equal acetylene/oxygen ratio and total flow rate reveals the similarity of the two flames. Although both spectra, shown in Figure 10, are quite simple compared to that of a nitrous oxide-acetylene flame, the background of the helium-oxygen-acetylene flame is about four times as intense as that of the air-acetylene flame at the strongest spectral feature observed (360 nm). However, this intensity ratio would be expected to equal 12.5 if only the temperature difference between the two flames were taken into consideration. This disparity indicates that a factor of three gain in elemental emission signal/background intensity should be observed for the $He-O_2-C_2H_2$ flame over the $air-C_2H_2$ flame. Studies are currently underway to substantiate this hypothesis.

CONCLUSIONS

With the development of a water-cooled burner, the helium-oxygen-acetylene

flame has been characterized to allow its safe and flashback-free use in
atomic spectrometry. The flame offers physical characteristics that are
similar to or better than the air-acetylene flame in the areas of temperature
and laminarity while yielding a slight disadvantage in background emission
intensity. It should offer increased sensitivity for the determination of
refractory elements while not possessing the high background and incidence
of ionization which plague the nitrous oxide-acetylene flame.

The incorporation of helium into the oxygen-acetylene flame gas mixture
has added a new control dimension to the flame. The third variable gas flow
enables the independent control of such flame properties as burning velocity,
temperature and rise velocity at any selected fuel/oxidant ratio. The flame
has a wide range of fuel/oxidant ratios available which should allow optimiza-
tion of flame conditions for many elements. In use, the operating costs of
the helium-containing flame exceed those of the air-acetylene flame, but are
comparable to the costs of a nitrous oxide-acetylene flame.

The analytical utility of the helium-oxygen-acetylene flame will be in-
vestigated by the measurement of atomization efficiencies, a project which is
currently in progress in our laboratories. The utility of the flame as an
atom source for atomic fluorescence will also be studied by means of fluores-
cence quantum efficiency measurements; we expect the measurements to substan-
tiate the importance of the flame's monatomic primary constituent to improved
fluorescence yields. Future work will also include the development of a flash-
back-free, air-cooled slot burner to support a long-path laminar $He-O_2-C_2H_2$
flame for use in atomic absorption spectrometry.

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CREDIT

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COOLING WATER

COPPER TUBING (5 mm i.d.)

BURNER TOP

BURNER BOWL

0.45 mm DIAMETER PORTS

SILVER SOLDER

3.5 cm

4.8 cm

BURNER

MIXING COIL

TOGGLE SHUT-OFF VALVES

NEEDLE VALVE

ROTAMETER

C_2H_2

O_2

He

FLASHBACK PREVENTED

FLASHBACK OCCURS

FUEL/OXIDANT RATIO

PER CENT HELIUM

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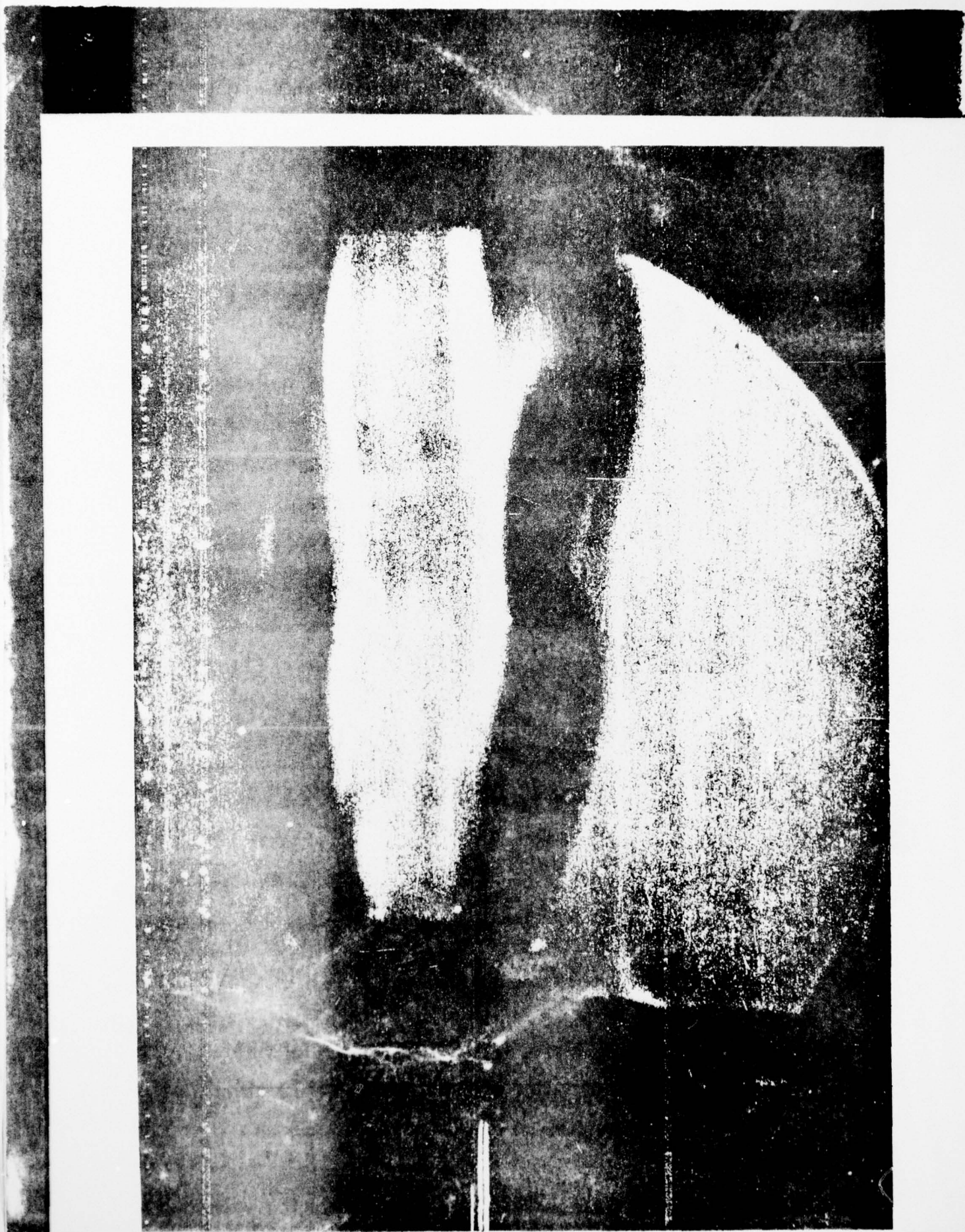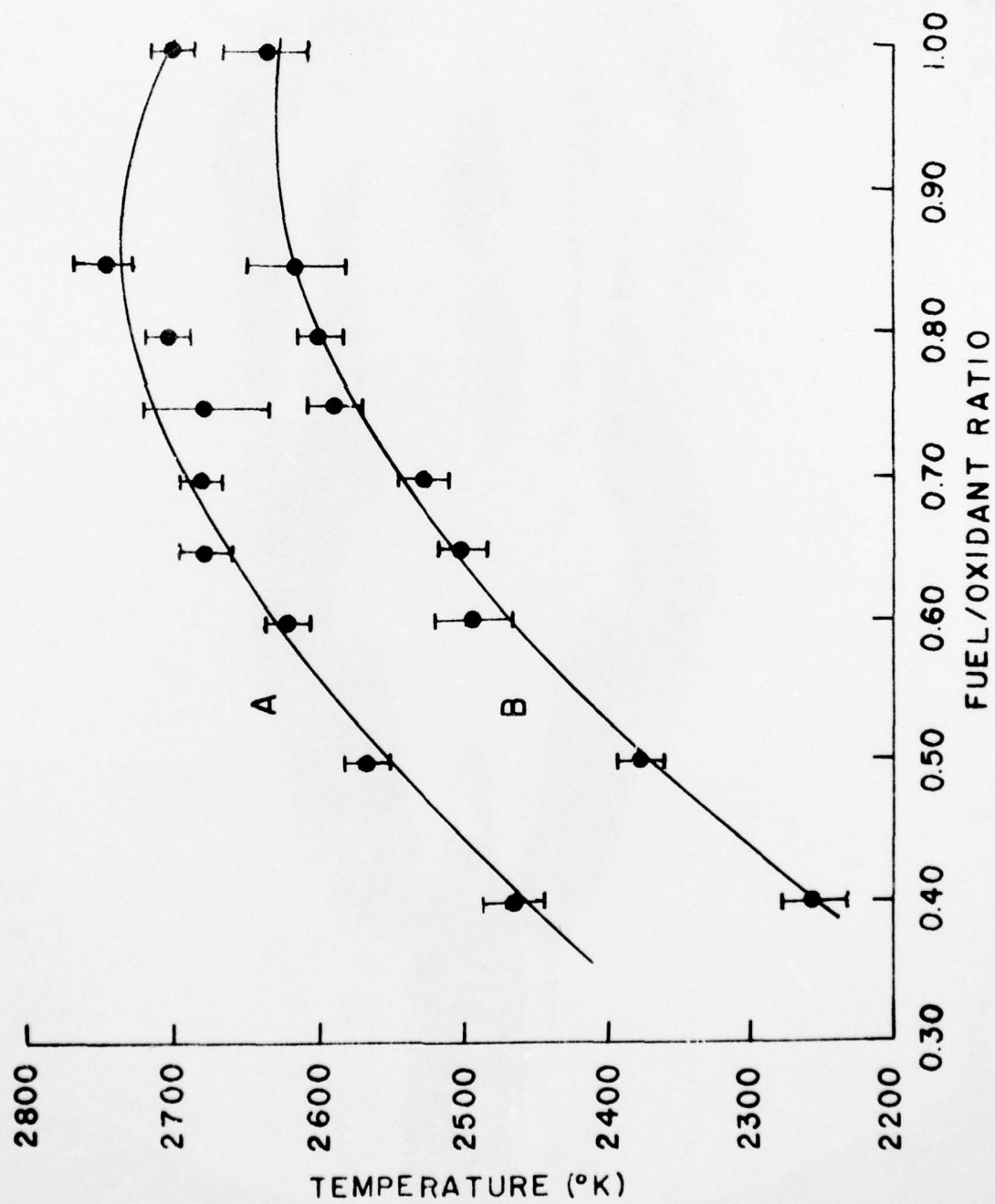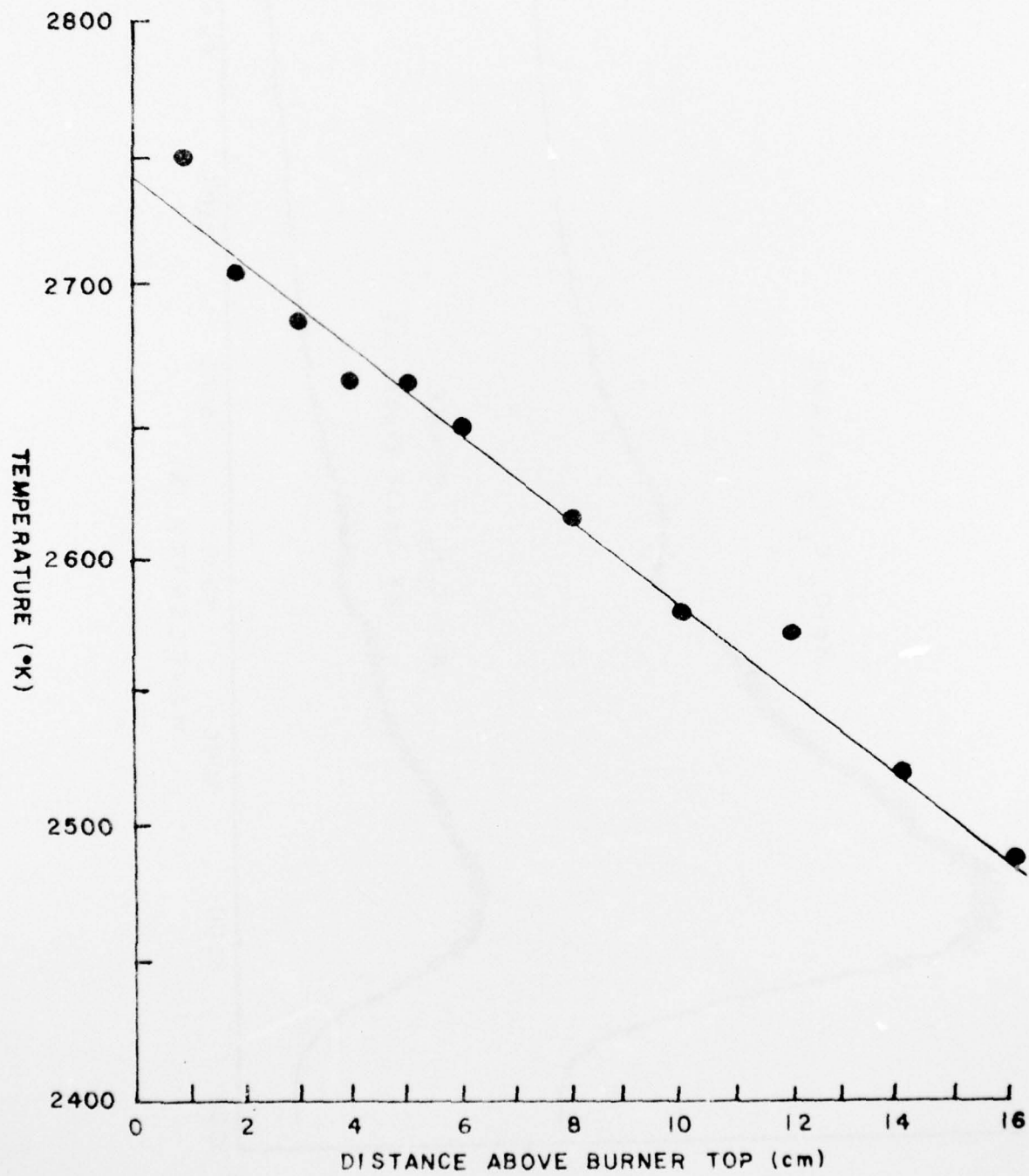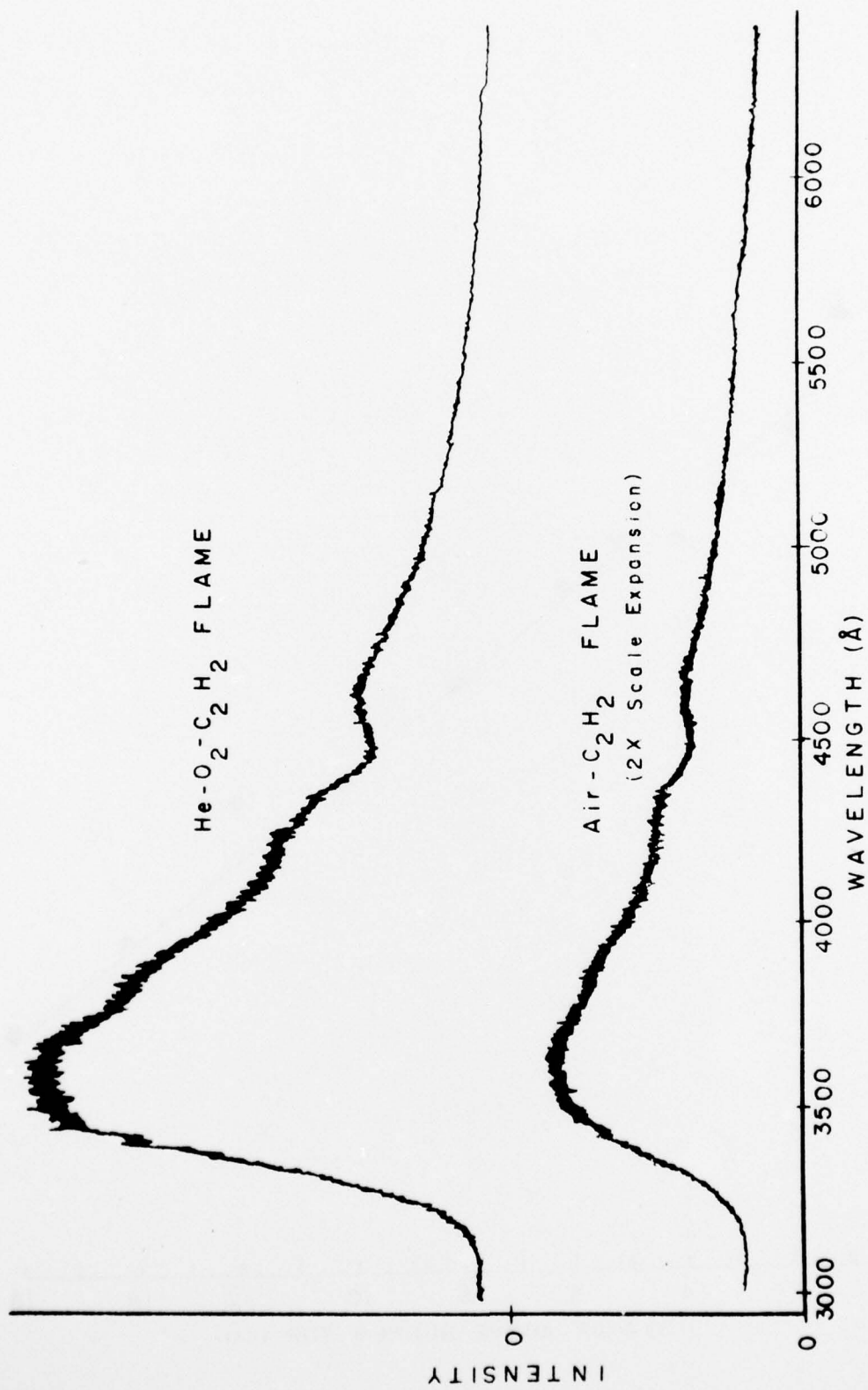   Attn: CRD-AA-IP
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China Lake, California 93555   1